

(19)



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(11) **EP 0 981 955 B1**

(12)

EUROPEAN PATENT SPECIFICATION

(45) Date of publication and mention
of the grant of the patent:
25.02.2004 Bulletin 2004/09

(51) Int Cl.7: **A01N 25/10, A01N 25/16,
A01N 53/00, A01N 51/00,
C08J 9/40, C08J 9/18,
C08J 9/06**

(21) Application number: **99306300.7**

(22) Date of filing: **10.08.1999**

(54) Impregnating polymer beads with insecticide

Imprägnieren von Polymer-Kügelchen mit Insektizid

Imprégnation des billes de polymère avec des insecticides

(84) Designated Contracting States:
**AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU
MC NL PT SE**

(56) References cited:
**EP-A- 0 200 197 EP-A- 0 281 918
US-A- 4 994 499**

(30) Priority: **19.08.1998 US 136243**

(43) Date of publication of application:
01.03.2000 Bulletin 2000/09

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- **PATENT ABSTRACTS OF JAPAN vol. 012, no. 433 (C-543), 15 November 1988 (1988-11-15) & JP 63 159451 A (SEKISUI PLASTICS CO LTD), 2 July 1988 (1988-07-02) & DATABASE WPI Derwent Publications Ltd., London, GB; AN 1988-224310 & JP 63 159451 A (SEKISUI PLASTICS CO. LTD.), 2 July 1988 (1988-07-02)**
- **PATENT ABSTRACTS OF JAPAN vol. 012, no. 421 (C-541), 8 November 1988 (1988-11-08) & JP 63 152648 A (NATL HOUSE IND CO LTD), 25 June 1988 (1988-06-25) & DATABASE WPI, 1988 Derwent Publications Ltd., London, GB; AN 1988-216722 & JP 63 152648 A (NATL. HOUSE IND. CO. LTD.), 25 June 1988 (1988-06-25)**

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Description

[0001] The present invention relates to polymers of vinyl aromatic monomers which contain insecticides. More particularly the present invention relates to foamable or expandable polymers of vinyl aromatic monomers which contain insecticides.

[0002] Polymeric foam is finding increasing application in the construction industry. However, under some conditions the foam may be subject to insect infestation, and particularly termite infestation. There is a need for methods of rendering polymeric foams resistant to insects.

[0003] United States Patent 5,194,323 issued March 16, 1993 and United States Patent 5,270,108 issued December 14, 1993, both assigned to AFM Corporation, disclose and claim polymeric foams suitable for construction purposes which have been treated with a borate compound to inhibit insect and, particularly, termite infestation. The patent does not teach or suggest that the insecticide could be added to the polymeric beads concurrently with the blowing agent.

[0004] United States Patent 5,704,172 issued January 6, 1998, assigned to The Dow Chemical Company teaches a rigid polymer foam having a plurality of grooves crossing in diagonal configuration which facilitates the application of insecticides to such rigid foam. The foam may be used for construction purposes. The patent teaches an external post fabrication application of insecticide and does not suggest that the insecticide may be incorporated into the polymer beads concurrently with the blowing agent.

[0005] Chemical Abstracts of Japanese Kokai 10036549 A2 published Feb. 10, 1998, and Japanese Kokai 63254143 published Oct. 20, 1988 teach applying anti-termite agents to the exterior of foams. The abstracts do not teach or suggest incorporating the insecticides into the polymer beads concurrently with impregnation with the blowing agent.

[0006] The Chemical Abstract of French Patent 2698632 published June 3, 1994, teaches the production of very light weight foams, preferably polyurethanes having a density from 5 to 20 g/l. While the patent teaches polyurethanes are preferred, it also teaches that the foam may comprise polystyrene, polyacrylates, and polycarbonates. The disclosure suggests the active substances are preferably introduced into the mixture before polymerization. However, examples 1 and 4 of the patent illustrate the invention using polystyrene foam. The active ingredient is not introduced into the monomer but rather is introduced into "a viscous but liquid styrene prepolymer which has been prepared by heating at 60°C for an unspecified period of time a mixture of 1000 g of styrene monomer, 1 g of benzoyl peroxide and 70 g of tributyl phosphate as a plastifier". MALATHION is added to the resulting prepolymer. The temperature is adjusted to 45°C and the mixture is stirred while nitrogen is bubbled through the mixture to

obtain a foam having a specific weight of 12 g/l. The patent neither teaches nor suggests that the active ingredient could be incorporated into polymer beads concurrently with impregnation with a blowing agent.

5 [0007] Chemical Abstract of Japanese Kokai 63264670 A2 published November 1, 1988 teaches impregnating polystyrene beads with boron compounds. The abstract of Japanese Kokai 63264670 A2 teaches that beads prepared by a bulk or emulsion polymerization are wetted with water and impregnated with boric acid. The abstract does not teach or suggest that the polymeric beads could be concurrently impregnated with blowing agent and insecticide.

10 [0008] Chemical Abstract of Japanese Kokai 63159451 A2 published July 2, 1988 teaches impregnating polystyrene beads with compounds selected from the group consisting of Phloxim, Fenitrothion, Cyanophos, Acephate and Prothiophos, concurrently with impregnation with a blowing agent. However, the active ingredients do not appear to be soluble in the blowing agent as the impregnation takes place using a solvent or an auxiliary solvent for the active ingredient together with the blowing agent. The present invention has eliminated the solvent or auxiliary solvent which is necessary in accordance with the Japanese Kokai.

15 [0009] Chemical Abstract of Japanese Kokai 63152648 teaches impregnating an expandable polystyrene bead with 2 ppm of Chlorpyrifos. This teaches away from the present invention as the bead is an expandable bead (i.e. already impregnated with blowing agent). Further the amount of insecticide is less than that of the present invention.

20 [0010] None of the above art discloses concurrently impregnating polymeric beads with blowing agent and an insecticide in the absence of a solvent.

25 [0011] Accordingly, one aspect of the present invention provides a process for incorporating an insecticide into an expandable bead of a thermoplastic polymer comprising impregnating said bead with from 100 to 10,000 ppm of said insecticide based on the weight of the thermoplastic concurrently with the impregnation of said bead with from 1 to 10 weight % based on the weight of said polymer of a blowing agent selected from the group consisting of C₄₋₆ alkanes.

30 [0012] The process of the present invention is carried out in the absence of any additional solvent or auxiliary solvent for the insecticide.

35 [0013] The thermoplastic of the present invention may comprise from 100 to 60 weight %, preferably from 100 to 80 weight % of one or more C₈₋₁₂ vinyl aromatic monomers and up to 40 weight %, preferably not more than 20 weight %, of other ethylenically unsaturated copolymerizable monomers. Examples of suitable vinyl aromatic monomers include, but are not limited to, styrene, alpha-methyl styrene, aromatic C₁₋₄alkyl substituted styrenes such as p-methyl styrene, p-ethyl styrene, p-isopropyl styrene, p-tert-butyl styrene and the like. Other ethylenically unsaturated copolymerizable mono-

mers may also be used including, for example, acrylic acid, methacrylic acid, maleic anhydride, methyl methacrylate, methyl acrylate, ethyl acrylate, butyl acrylate, acrylonitrile, methacrylonitrile and the like.

[0014] A particularly useful thermoplastic is polystyrene in which the monomer is 100% styrene.

[0015] The insecticide may be incorporated into beads of the polymer in an amount from 100 to 10,000 parts per million (ppm) (corresponding to from 0.01 to 1 weight %), preferably from 300 to 5,000 ppm (corresponding to from 0.03 to 0.5 weight %), based on the weight of the polymer.

[0016] While a number of insecticides are available, some useful insecticides may be selected from the group consisting of 1-[(6-chloro-3-pyridinyl)methyl]-4,5-dihydro-N-nirto-1H-imidazol-2-amine and 3-(2,2-dichloroethenyl)-2,2-di-methylcyclopropanecarboxylic acid cyano(3-phenoxyphenyl)-methyl ester (cypermethrin), the active ingredient in, for example, Demon TC sold by Zeneca; 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylic acid (3-phenoxyphenyl)methyl ester (permethrin), the active ingredient in, for example, Dragnet FT and Torpedo sold by Zeneca; and 1-[(6-chloro-3-pyridinyl)methyl]-4,5-dihydro-N-nirto-1H-imidazol-2-amine (imidacloprid) the active ingredient in, for example, Premise sold by Bayer. The insecticide should be transported into the polymer bead together with the blowing agent. Preferably the insecticide should be at least moderately soluble in the blowing agent.

[0017] The thermoplastic beads of the present invention may be polymerized in a conventional suspension or emulsion polymerization. Generally, thermoplastics comprising a major amount of a vinyl aromatic monomer and a minor amount of one or more copolymerizable monomers may be polymerized using a thermal and/or free radical initiation. The monomer(s) is/are suspended or dispersed in a different, non-hydrocarbon, typically aqueous, phase and the polymerization takes place in the dispersed monomer droplets (e.g. suspension) or in a micelle into which monomer diffuses from the monomer droplets (e.g. emulsion).

[0018] The monomers are suspended in water from about 50 to 500 parts (preferably, about 75 to 250 parts) by weight per 100 parts by weight of the monomers using an effective amount of one or more suitable suspending agents. Any of the suspending agents useful in the suspension polymerization of vinyl aromatic polymers may be used. Examples of suitable suspending agents include finely divided water-insoluble inorganic substances such as tricalcium phosphate, and the like as well as water-soluble polymers such as polyvinyl alcohol, alkyl aryl sulfonates, hydroxyethyl cellulose, polyacrylic acid, methyl cellulose, polyvinyl pyrrolidone, and low molecular weight (preferably having an M_n less than about 5,000 polyalkylene glycols (e.g. polyethylene glycols and polypropylene glycols) and the like. Auxiliary suspending agents such as sodium linear alkylbenzene

sulfonates may also be employed. The use of tricalcium phosphate together with a sodium linear alkylbenzene sulfonate is particularly useful. The amount of the suspending agent necessary will vary depending on a number of factors but will generally be from about 0.01 to 1 part by weight per 100 parts by weight of the vinyl aromatic polymer. One or more surfactants such as a polyoxyalkylene derivative of sorbitan monolaurate or other fatty acid ester, an ethylene oxide/propylene oxide block copolymer, or other non-ionic or anionic surface active agent can be added to the aqueous suspension if desired. The preferred amount of surfactant is from about 0.01 to 1 part by weight per 100 parts by weight of monomer.

[0019] In addition to the monomers, the aqueous suspension may further include a free radical initiator or free radical initiator system. The free radical generator may be a peroxide such as hydrogen peroxide or benzoyl peroxide, or a persulfate initiator.

[0020] The reaction mixture is heated to initiate polymerization, either thermally or by a free radical catalyst. After the monomers are polymerized to form particles or beads (generally resulting from the suspension process) or microparticles (generally resulting from the emulsion process), they may be separated from the aqueous phase and washed. The thermoplastic polymer beads are typically from about 0.1 to 2 mm in average diameter.

[0021] Generally, the polymer bead is impregnated with a blowing agent to make expandable thermoplastic beads (polystyrene). Typically the beads may be impregnated with from 1 to 10 weight %, preferably from about 3 to 8 weight %, based on the weight of the polymer of one or more blowing agents selected from the group consisting of C_{4-6} alkanes. Typical blowing agents include butane, pentane and hexane. While the CFC's and HCFC's such as dichlorodifluoromethane, trichlorofluoromethane and dichlorofluoromethane have some suitable properties as blowing agents, their use is not recommended.

[0022] In the suspension process the polymer beads may be impregnated either concurrently with the later part of the polymerization or after polymerization. In the impregnation process the blowing agent, together with the insecticide, are added to an aqueous suspension containing polymeric beads. The suspensions, together with the blowing agent and the insecticide, are maintained at a temperature typically from about 60°C to 110°C for a period of time from about 2 to 4 hours by which time the blowing agent and insecticide have impregnated the polymeric beads. The beads are then separated from the emulsion, optionally washed and dried.

[0023] The resulting beads are stored preferably in a cool dark environment. When used to produce a foam product, typically the beads are first partially expanded and then placed in a mold and fully expanded resulting in a fusion of the foamed beads and a closed cell foam.

Generally, for construction industry purposes, the foam will take the form of a sheet.

[0024] The present invention will now be illustrated by the following examples. In the examples unless otherwise indicated, parts means parts by weight (e.g. grams) and per cent means weight per cent.

[0025] In a preferred embodiment of the present invention the polymer may further incorporate a flame retardant. Typically the flame retardant is incorporated into the polymer in an amount from 5,000 ppm to 50,000 ppm (0.5 weight % to 5 weight %), preferably from 7,500 ppm to 15,000 ppm. In the suspension or emulsion process the flame retardant may be added to the monomer or aqueous phase, depending on solubility in the monomer/polymer. In the bulk process and, optionally, in the suspension and emulsion process, the flame retardant may be added to the resulting polymer by coating it or adding it as part of an additive package in an extrusion process. Suitable flame retardants are known to those skilled in the art. Some flame retardants include hexabromocyclododecane, dibromoethyldibromocyclohexane, tetrabromocyclooctane, tribromophenol alkyl ether, tetrabromobisphenol A - bis (2,3-dibromopropyl ether).

Experiment 1

[0026] Styrene monomer was suspended in an aqueous phase in the presence of 0.20 weight % of a primary inorganic suspending agent and 0.25 weight % of a secondary anionic surfactant suspending agent, based on the weight of the styrene monomer. Low and high temperature peroxide initiators were added at levels of 0.34 and 0.066 weight %, respectively. Nucleation agents were also added at levels of 0.2 weight %. The resulting suspension was heated to 90°C and the first phase of the polymerization of styrene monomer to polymer was carried out over 5.5 hours. The suspension was then heated from 90-115°C and the second phase of the polymerization was carried out over 2 hours. The resulting beads were washed, dried and re-suspended in water with 0.6 weight % primary suspending agent. Imidacloprid was added to the suspension at 0.03 weight % based on the weight of the polymer in the absence of any additional solvent for the imidacloprid. The suspension was heated from 70-115°C over 2.5 hours as 7.2 weight % of pentane was added to the system. The system was held at 115°C for 1.5 hours to fully impregnate the polystyrene beads with the blowing agent and the imidacloprid. The resulting beads were then washed, dried and lubed with 0.15 weight % stearate.

[0027] The resulting beads were then partially expanded in steam. After aging, the pre-expanded beads were placed in a closed mold and heated to 115°C to fully expand and fuse them together. The result is a sheet or shape of foam. No negative performance characteristics were observed in the blowing of the foam.

[0028] It should be noted that the insecticide imparted

a light brown tint to the impregnated beads.

Experiment 2

[0029] The procedure of experiment 1 was repeated, except that the amount of imidacloprid was 0.3 weight % based on the weight of the polymer. The polymer was again tinted a light brown. Again there was no negative performance characteristics observed in foaming the beads.

Experiment 3

[0030] The procedure of experiment 1 was repeated except that cypermethrin was used at a level of 0.3 weight % based on the weight of the polymer. Again there were no negative performance characteristics observed in foaming the beads.

Claims

1. A process for incorporating an insecticide into an expandable bead of a thermoplastic polymer comprising impregnating said bead with from 100 to 10,000 ppm of said insecticide based on the weight of the thermoplastic concurrently with the impregnation of said bead with from 1 to 10 weight % based on the weight of said polymer of a blowing agent selected from the group consisting of C₄₋₆ alkanes, characterised in that impregnation of said bead with said insecticide is in the absence of any additional solvent for such insecticide.
2. The process according to claim 1, wherein said insecticide is selected from the group consisting of 1-[(6-chloro-3-pyridinyl) methyl]-4, 5- dihydro-N-nitro-1H-imidazol-2-amine; 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylic acid cyano (3-phenoxyphenyl)-methyl ester; and 3-(2,2 dichloroethenyl)-2,2-dimethylcyclopropanecarboxylic acid (3- phenoxyphenyl) methyl ester.
3. The process according to claim 2, wherein said one or more monomers is selected from the group consisting of C₈₋₁₂ vinyl aromatic monomers; C₁₋₄ alkyl esters of acrylic or methacrylic acid, acrylonitrile, and metacrylonitrile.
4. The process according to claim 3, wherein said thermoplastic bead is prepared by an emulsion polymerization.
5. The process according to claim 4, wherein said blowing agent is one or more compounds selected from the group consisting of butane, pentane and hexane.

6. The process according to claim 5, wherein said monomer is a C₈₋₁₂ vinyl aromatic monomer.
7. The process according to claim 6, wherein said monomer is selected from the group consisting of styrene, alpha-methyl styrene, and p-methyl styrene.
8. The process according to claim 7, wherein said insecticide is present in an amount from 300 to 5000 ppm based on the weight of the monomer.
9. The process according to claim 8, wherein said insecticide is 1-[(6-chloro-3-pyridinyl)methyl]-4,5-dihydro-N-nitro-1H-imidazol-2-amine.
10. The process according to claim 8, wherein said insecticide is 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylic acid cyano(3-phenoxyphenyl)-methyl ester.
11. The process according to claim 8, wherein said insecticide is 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylic acid (3-phenoxyphenyl)methyl ester.
12. The process according to claim 9, claim 10 or claim 11 wherein said monomer is styrene.
13. The process according to claim 12, further including adding from 5,000 ppm to 50,000 ppm of a flame retardant into said polymer.
14. The process according to claim 13, wherein said flame retardant is selected from the group consisting of hexabromocyclododecane, dibromoethyldibromocyclohexane, tetrabromocyclooctane, tribromophenol alkyl ether, tetrabromobisphenol A-bis (2,3-dibromopropyl ether).

Patentansprüche

1. Verfahren zum Einbauen eines Insektizids in eine schäumbare Perle aus einem thermoplastischen Polymer, umfassend das Imprägnieren der Perle mit 100 bis 10.000 ppm des Insektizids, bezogen auf das Gewicht des Thermoplasten, gleichzeitig mit dem Imprägnieren der Perle mit 1 bis 10 Gew.-%, bezogen auf das Gewicht des Polymers, eines Treibmittels, das aus der aus C₄₋₆-Alkanen bestehenden Gruppe ausgewählt ist, dadurch gekennzeichnet, dass das Imprägnieren der Perle mit dem Insektizid in Abwesenheit jeglichen zusätzlichen Lösungsmittels für ein derartiges Insektizid erfolgt.
2. Verfahren nach Anspruch 1, worin das Insektizid aus der aus 1-[(2-Chlor-3-pyridinyl)methyl]-4,5-di-

hydro-N-nitro-1H-imidazol-2-amin; 3-(2,2-Dichlorethenyl)-2,2-dimethylcyclopropanecarbonsäure-cyano-(3-phenoxyphenyl)methylester; und 3-(2,2-Dichlorethenyl)-2,2-dimethylcyclopropanecarbonsäure-(3-phenoxyphenyl)methylester bestehenden Gruppe ausgewählt ist.

3. Verfahren nach Anspruch 2, worin das eine oder die mehreren Monomer(e) aus der aus C₈₋₁₂-Vinylaromaten-Monomeren; C₁₋₄-Alkylestern von Acryl- oder Methacrylsäure, Acrylnitril und Methacrylnitril bestehenden Gruppe ausgewählt ist bzw. sind.
4. Verfahren nach Anspruch 3, worin die thermoplastische Perle durch Emulsionspolymerisation hergestellt wird.
5. Verfahren nach Anspruch 4, worin das Treibmittel aus einer oder mehreren Verbindung(en), ausgewählt aus der aus Butan, Pentan und Hexan bestehenden Gruppe, besteht.
6. Verfahren nach Anspruch 5, worin das Monomer ein C₈₋₁₂-Vinylaromaten-Monomer ist.
7. Verfahren nach Anspruch 6, worin das Monomer aus der aus Styrol, α-Methylstyrol und p-Methylstyrol bestehenden Gruppe ausgewählt ist.
8. Verfahren nach Anspruch 7, worin das Insektizid in einer Menge von 300 bis 5.000 ppm, bezogen auf das Gewicht des Monomers, vorhanden ist.
9. Verfahren nach Anspruch 8, worin das Insektizid 1-[(6-Chlor-3-pyridinyl)methyl]-4,5-dihydro-N-nitro-1H-imidazol-2-amin ist.
10. Verfahren nach Anspruch 8, worin das Insektizid 3-(2,2-Dichlorethenyl)-2,2-dimethylcyclopropanecarbonsäure-cyano-(3-phenoxyphenyl)methylester ist.
11. Verfahren nach Anspruch 8, worin das Insektizid 3-(2,2-Dichlorethenyl)-2,2-dimethylcyclopropanecarbonsäure-(3-phenoxyphenyl)methylester ist.
12. Verfahren nach Anspruch 9, 10 oder 11, worin das Monomer Styrol ist.
13. Verfahren nach Anspruch 12, das weiters den Zusatz von 5.000 ppm bis 50.000 ppm eines Flamm-schutzmittels in das Polymer umfasst.
14. Verfahren nach Anspruch 13, worin das Flamm-schutzmittel aus der aus Hexabromcyclododecan, Dibromethyldibromcyclohexan, Tetrabromcyclooctan, Tribromphenolalkylether, Tetrabrombisphenol-A-bis-(2,3-dibrompropylether) bestehenden Grup-

pe ausgewählt ist.

clopropanecarboxylique cyano(3-phénoxyphényl)-méthyl ester.

Revendications

1. Procédé pour incorporer un insecticide dans une bille dilatable d'un polymère thermoplastique comprenant l'imprégnation de ladite bille de 100 à 10 000 ppm dudit insecticide en se basant sur le poids du thermoplastique, concurremment avec l'imprégnation de ladite bille de 1 à 10% en poids en se basant sur le poids dudit polymère d'un agent gonflant sélectionné dans le groupe consistant en alcanes C₄₋₆, caractérisé en ce que l'imprégnation de ladite bille par ledit insecticide est en l'absence de tout solvant additionnel pour un tel insecticide. 5
2. Procédé selon la revendication 1, où ledit insecticide est sélectionné dans le groupe consistant en 1-[(6-chloro-3-pyridinyl)méthyl]-4, 5-dihydro-N-nitro-1H-imidazole-2-amine; acide 3-(2,2-dichloroéthényl)-2,2-diméthylcyclopropanecarboxylique cyano (3-phénoxyphényl)-méthyl ester; et acide 3-(2,2-dichloroéthényl)-2,2-diméthylcyclopropanecarboxylique (3-phénoxyphényl) méthyl ester. 10 20 25
3. Procédé selon la revendication 2, où ledit un ou plusieurs monomères est sélectionné dans le groupe consistant en monomères vinyle aromatiques C₈₋₁₂; alkyl esters C₁₋₄ d'acide acrylique ou méthacrylique, acrylonitrile, et méthacrylonitrile. 30
4. Procédé selon la revendication 3, où ladite bille thermoplastique est préparée par une polymérisation en émulsion. 35
5. Procédé selon la revendication 4, où ledit agent gonflant est un ou plusieurs composés sélectionnés dans le groupe consistant en butane, pentane et hexane. 40
6. Procédé selon la revendication 5, où ledit monomère est un monomère vinyle aromatique C₈₋₁₂. 45
7. Procédé selon la revendication 6 où ledit monomère est sélectionné dans le groupe consistant en styrène, alpha-méthyl styrène, et p-méthyl styrène. 50
8. Procédé selon la revendication 7, où ledit insecticide est présent en une quantité de 300 à 5000 ppm en se basant sur le poids du monomère. 55
9. Procédé selon la revendication 8, où ledit insecticide est la 1[(6-chloro-3-pyridinyl)méthyl]-4,5-dihydro-N-nitro-1H-imidazole-2-amine. 55
10. Procédé selon la revendication 8, où ledit insecticide est l'acide 3-(2,2-dichloroéthyl)-2,2-diméthylcy- 5
11. Procédé selon la revendication 8, où ledit insecticide est l'acide 3-(2,2-dichloroéthyl)-2,2-diméthylcyclopropanecarboxylique (3-phénoxyphényl)méthyl ester. 10
12. Procédé selon la revendication 9, la revendication 10 ou la revendication 11 où ledit monomère est du styrène. 15
13. Procédé selon la revendication 12, comprenant de plus l'addition de 5000 ppm à 50 000 ppm d'un ignifuge dans ledit polymère. 20
14. Procédé selon la revendication 13, où ledit ignifuge est sélectionné dans le groupe consistant en hexabromocyclododécane, dibromoéthylidibromocyclohexane, tétrabromocyclooctane, tribromophénol alkyl ester, tétrabromobisophénol A-bis (2,3-dibromopropyl éther). 25